Flexural Properties of Macadamia Nutshell Particle Reinforced Polyester Composites

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Abstract

A study on the flexural properties of macadamia nutshell particle reinforced polyester composites is presented in this paper. The macadamia nutshell/polyester composites were made as per the hand layup procedure and the instructions provided by the manufacturers. These specimens were then tested in the three point bend configuration in accordance with ASTM D790-07 at a span to depth ratio of 16. Four weight fractions of macadamia nutshell particles 10%, 20%, 30% and 40% were chosen to be studied. The process-induced voids were studied and it is shown that voids play an important role in flexural properties. When voids exist, flexural modulus was calculated using a micromechanical model. It is shown from both the experimental results and calculation that flexural modulus increases with the weigh fraction of macadamia nutshell particles, while decreases with increasing void content. Adding macadamia nutshell particles does not improve the flexural strength of polyester. It is shown flexural strength decreases with increasing void content.

Keywords: Polymer-matrix composites (PMCs); Macadamia nutshell; Flexural; Mechanical testing

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1 Introduction

The interest in natural fiber reinforced biocomposites has been growing rapidly during recent years, driven by increased environmental and health concerns, more sustainable methods of manufacture and reduced energy consumption. Natural fibers such as cotton, flax and hemp have been used as reinforcements in biocomposites [1]. Waste generation is a significant burden on the environment. Most food and forestry industry activities result in large amounts of by-products that are often treated as waste and sent to landfill. Macadamia is a genus of flowering plants in the family of Proteaceae, with seven species native to Australia. The shells and other waste comprise almost 70% by weight of the macadamia nuts. They can be burned as a wood substitute in coffee roasting, ground to produce organic waste for gardening, used for mulch in the nut tree orchards, or used for chicken litter that, after use, returns to the orchard for use as fertilizer [2].

Macadamia nutshell is hard and brittle. As a biological structure, it is highly optimized and efficient in terms of strength and toughness due to ecological evolution and selection [3]. It is known to have approximately the same fracture toughness as common ceramics and glass, and when compared on the basis of specific strength or modulus, it outperforms these materials due to its low density. Macadamia nutshell is a cellular solid with relatively low density and high strength, but its structure is reasonably isotropic and uniform, very different from that of trees.

It is shown from the literature that the research on macadamia nutshell reinforced composites is quite limited. Husque has been making products from pulverized macadamia nutshell bonded with resin [4, 5].

In this study, the flexural properties of macadamia nutshell particle reinforced polyester composites are studied through a three-point bend test, in which a loading nose deflects a specimen at a set span and loading rate until fracture. A fixed span-to-depth ratio 16 was used and four weight fractions of macadamia nutshell particles 10%, 20%, 30% and 40% were chosen to be studied. The process-induced voids were also studied.

2 Experiments

The materials used in this study were macadamia nutshell (M. integrifolia) with husk removed (MacNuts WA), SR250 orthophthalic polyester resin and MEKP catalyst (Fibreglass & Resin Sales Pty. Ltd.) Their properties are shown in Table 1 [6]. Four weight fractions of macadamia nutshell particles 10%, 20%, 30% and 40% were chosen to be studied. For reference, pure polyester specimens were also made and tested.

The macadamia nutshell as purchased was first broken into small pieces to fit in a ROCKLABS standard ring mill pulverizer as shown in Figure 1. These nutshell pieces were then pulverized into fine powder as shown in Figure 2. The macadamia nutshell/polyester composites were made as per the hand layup procedure [7] and the instructions provided by the manufacturers. The mold being used in this study consists of a flat tray and a top plate. Prior to manufacturing, the mold surfaces were prepared by applying a layer of traffic wax and letting it dry for 20 to 25 minutes.

The required amount of unsaturated polyester resin and macadamia nutshell powder was measured in a mixing cup and in the bottom mold, respectively. The suitable amount of MEKP catalyst was calculated according to the instruction provided by the manufacturer, i.e. 10ml of MEKP per kilogram of resin, and then added into the polyester resin. The polyester resin and MEKP catalyst were mixed for 2 minutes using the scrap-rotate-scrap mixing method as suggested by the manufacturer, where the sides and bottom of the mixing cup were scrapped in care by using a pop-stick to reduce trapping of air bubbles, while 90° rotations were made.

The catalyzed polyester resin was then poured slowly into the tray at a distance of 30 cm above the tray, allowing extremely thin pouring stream to reduce trapping of air bubbles. The scrap-rotate-scrap method was again applied for 2 to 5 minutes or longer depending on the composition of macadamia nutshell powder. The mixture was left untouched for 10 minutes to allow air bubbles to rise to the surface, and pop-stick was used to "pop" the bubbles.

The top plate was stacked onto the gel-like mixture and the mixture was left for 24 hours for curing. The composite was then post-cured for 2 hours at 80°C before being removed from the mold.

The finished composites were cut into test specimens of 25mm wide using a diamond tipped circular saw blade. Testing was conducted in a three point bend configuration in accordance to procedure A of ASTM D790-07, using an Instron 550R universal testing machine at a span-to-depth ratio of 16, as shown in Figure 3. The average loading rate for this analysis was in the order of 5 mm/min. For each specimen, its length, width, thickness and weight were measured in order to determine the density.

From the load-deflection data obtained from testing, flexural strength (σ_F), modulus (E_F) and strain to failure (ε_F) can be calculated by [8]:

$$\sigma_F = \frac{3P_{max}L}{2bh^2}\left[1 + 6\left(\frac{D}{L}\right)^2 - 4\left(\frac{h}{L}\right)\left(\frac{D}{L}\right)\right] \tag{1}$$

$$E_F = \frac{mL^3}{4bh^3} \tag{2}$$

$$\varepsilon_F = \frac{6Dh}{L^2} \tag{3}$$

where L, b and h are the span, width and depth of the specimen, m is the slope of the tangent to the initial straight-line portion of the load-deflection curve, D is the maximum deflection before failure, and P_{max} is the maximum load encountered before failure.

For each weight loading, 5 tests were conducted for each configuration, and the average values and variations were determined and presented. Following the testing, specimens were also inspected under an optical microscope in order to investigate any anomalies in flexural performance.

3 Results and Discussion

3.1 Fracture Surface

The micrograph images of representative failed specimens are shown in Figure 4. It is seen that all test specimens failed in a brittle manner.

3.2 Load-Displacement Curve

The load-displacement curves from testing are shown in Figure 5. It is seen that load increases linearly with displacement until the failure point. It is shown that for some cases, e.g. 20% nutshell, the test curves have a wide spread.

3.3 Void Content

When voids are present, the density of composite can be obtained using the rule of mixtures as given below.

$$\rho_c = \rho_p V_p + \rho_m \left(1 - V_p - V_v\right) \tag{4}$$

where ρ_c, ρ_p and ρ_m are the densities of composite, reinforcement and matrix, respectively, and V_p and V_v are the volume fractions of reinforcing particles and voids, respectively.

In this study, the void contents in the pure polyester specimens were unknown. Thus, the pure polyester specimens were regarded as void-free and the voids caused by the introduction of macadamia nutshell particles were studied. Since the densities of macadamia nutshell and polyester are close, the void content is determined by

$$V_v = 1 - \frac{\rho_c}{\rho_m} \tag{5}$$

The densities and the void contents obtained are shown in Table 2. It is seen that void content decreases with increasing weight percentage of macadamia nutshell particles and the specimens consisting of 10% macadamia nutshell particles have the highest void content.

3.4 Flexural Modulus

Since the macadamia nutshell/polyester composites contain voids, they are three-phase materials and modulus is given by [9]

$$E = E_m \left(\frac{1 - V_v^{2/3}}{1 - V_v^{2/3} + V_v} \right) \left[\frac{1 + \left(E_p / E_m - 1 \right) V_p^{2/3}}{1 + \left(E_p / E_m - 1 \right) \left(V_p^{2/3} - V_p \right)} \right] \tag{6}$$

where E, E_p and E_m are the elastic moduli of the composite, macadamia nutshell particles and polyester, respectively.

Because of the linear load-displacement relationships, the elastic modulus and the flexural modulus are very close. The flexural moduli from the experiments and calculation are shown in Figure 6. It is seen that good agreement is found. The general trend is flexural modulus increases with weight percentage of macadamia nutshell particles except the 10% macadamia nutshell specimens, which have the lowest flexural modulus because of their highest void content.

3.5 Flexural Strength

The flexural strengths from the experiments are shown in Figure 7. No clear trend can be seen. It is shown from Table 1 that macadamia nutshell and polyester have similar strengths. Thus, it is inferred that flexural strength is affected by void content. A plot of flexural strength vs. void content is given in Figure 8. It is seen that flexural strength decreases with increasing void content. The relationship between flexural strength and void content can be given by an empirical formula [10] which reads

$$\sigma_F = 46.08 V_v^{-0.4585} \tag{7}$$

where V_v is the percentage of voids.

It is shown that the introduction of macadamia nutshell particles does not reinforce polyester. The reason is possibly hydrophilic macadamia nutshell is incompatible with hydrophobic polyester. This will result in poor interfacial adhesion.

3.6 Strain to Failure

The strains to failure from the experiments are shown in Figure 9. It is seen that the trend is similar to that of the flexural strength. A plot of strain to failure vs. void content is given in Figure 10. It is shown that strain to failure is strongly dependent on void content.

4 Conclusions

The flexural properties of macadamia nutshell particle reinforced polyester were studied. Tests were conducted in the three point bend configuration in accordance with ASTM D790-07 at a span to depth ratio of 16. Four weight fractions of macadamia nutshell particles 10%, 20%, 30% and 40% were chosen to be studied. The process-induced voids were studied and it is shown that voids play an important role in flexural properties. When voids exist, flexural modulus was calculated using a micromechanical model. It is shown from both the experimental results and calculation that flexural modulus increases with the weigh fraction of macadamia nutshell particles, while decreases with increasing void content. Adding macadamia nutshell particles does not improve the flexural strength of polyester. It is shown flexural strength decreases with increasing void content.

Acknowledgment

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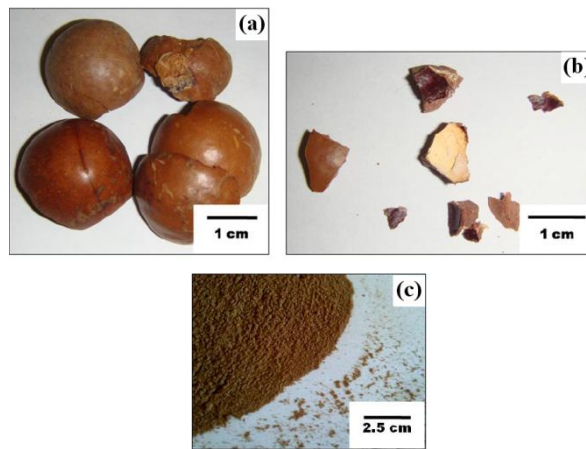**Figure 1: ROCKLABS standard ring mill pulverizer**

Figure 2: (a) Size of macadamia nutshell purchased from the market; (b) Preferable nutshell size filled into ring meal head; (c) Macadamia nutshell powder after ring mill pulverizing
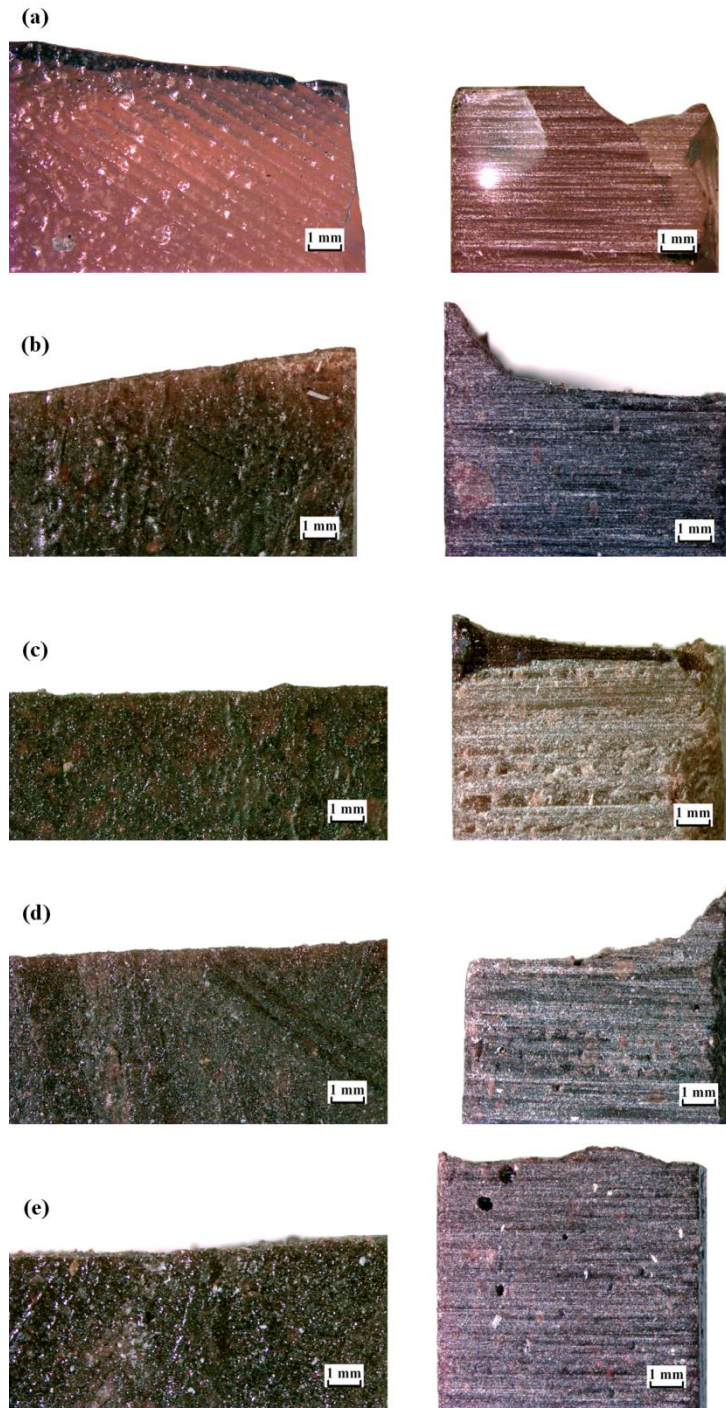

Figure 3: Flexural testing being conducted

Figure 4: Micrograph images of failed specimens (a) pure polyester (b) 10% nutshell (c) 20% nutshell (d) 30% nutshell (e) 40% nutshell
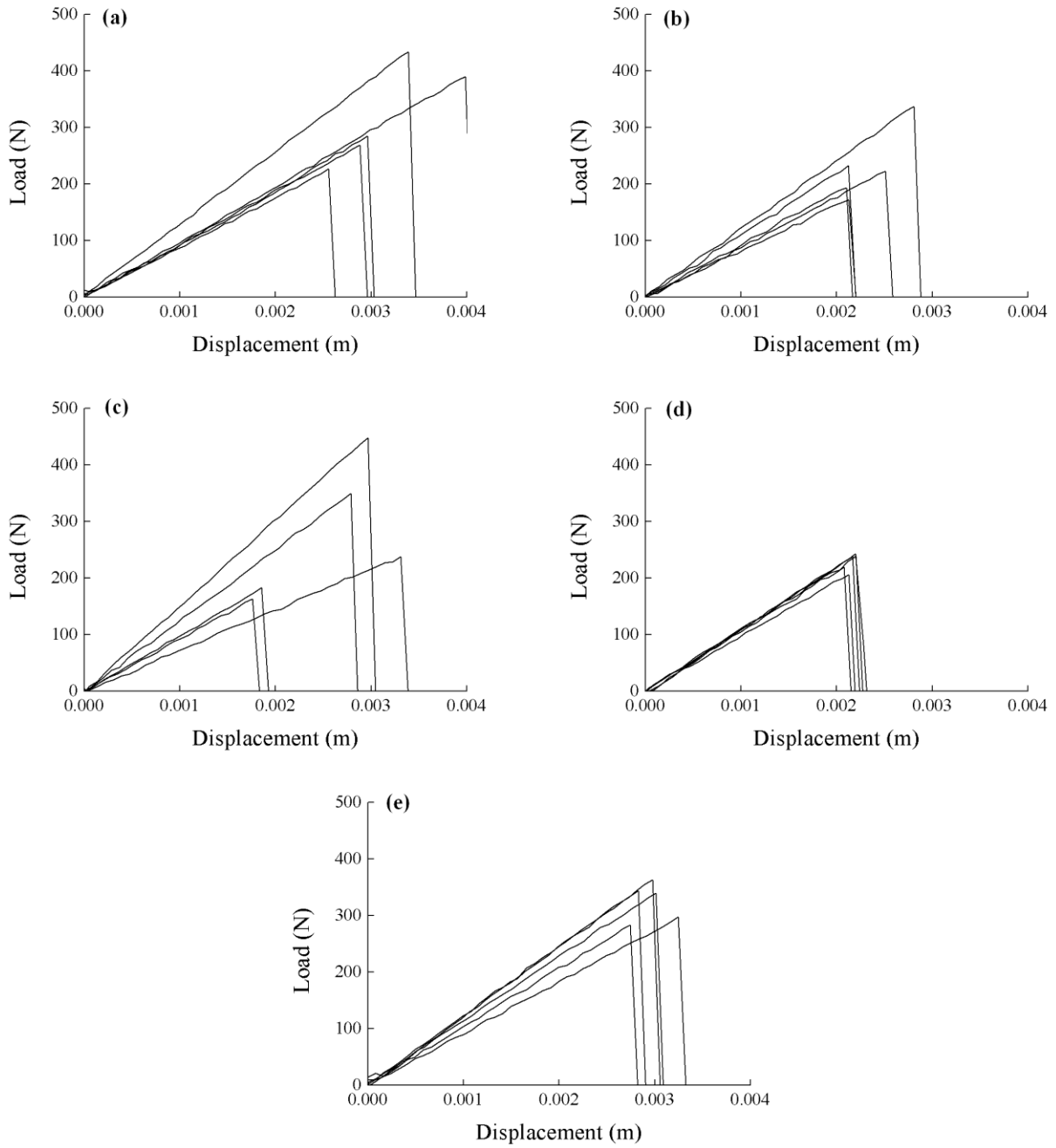
Figure 5: Load-displacement curves from flexural tests (a) pure polyester (b) 10% nutshell (c) 20% nutshell (d) 30% nutshell (e) 40% nutshell
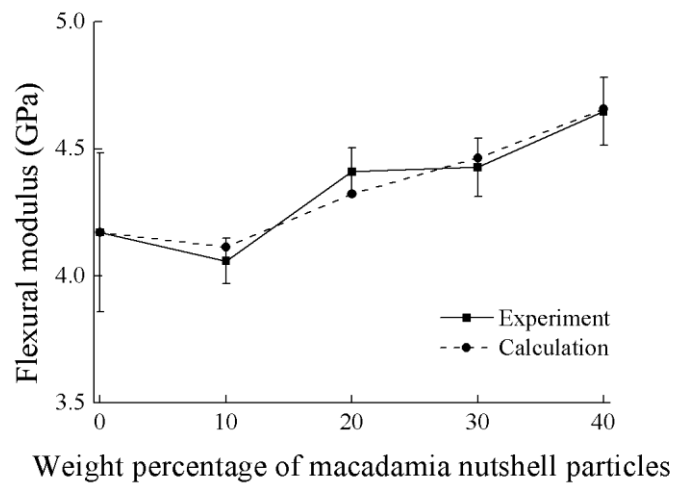
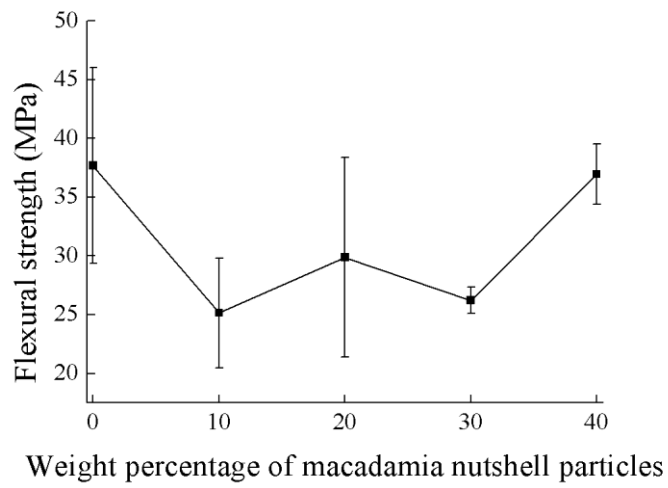**Figure 6: Flexural moduli from experiments and calculation**

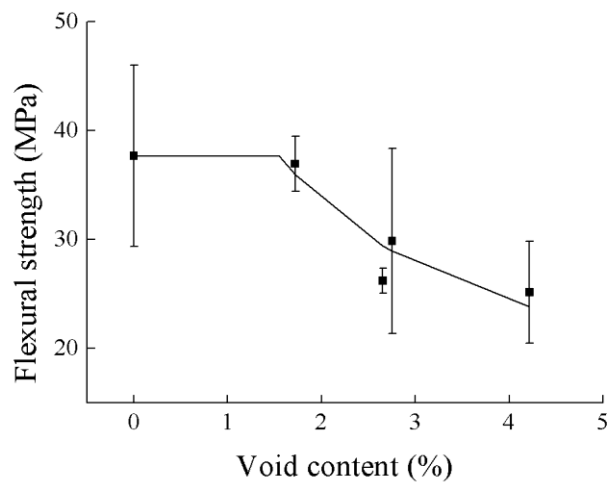**Figure 7: Flexural strength vs. weight percentage of macadamia nutshell particles**

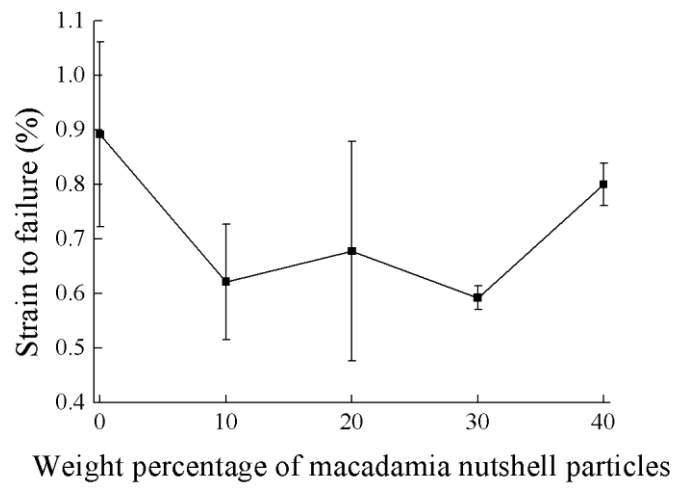**Figure 8: Flexural strength vs. void content**

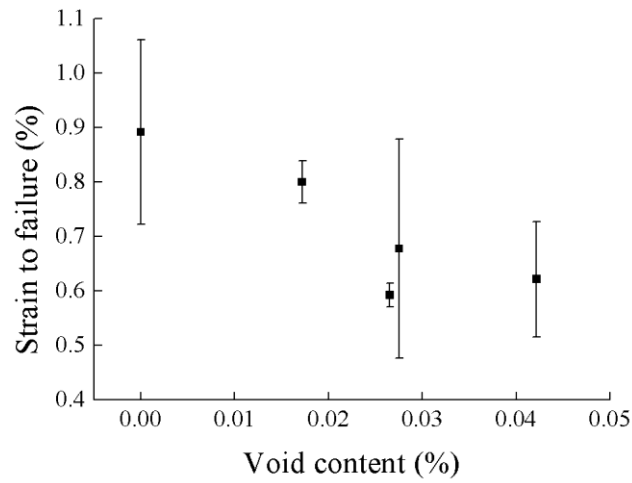**Figure 9: Strains to failure vs. weight percentage of macadamia nutshell particles**

Figure 10: Strains to failure vs. void content

Table 1: Constituent materials and selected properties

Material	Tensile strength (MPa)	Tensile modulus (GPa)	Density (g/cm^3)
Macadamia nutshell	56	5.7	1.27
Polyester	52	4.0	1.1-1.4

Table 2: Measured thicknesses, densities and void contents

Weight percentage of macadamia nutshell particles	Average thickness (mm)	Measured density (g/cm^3)	Void content (%)
0	8.20	1.260	0
10	8.80	1.207	4.22
20	8.71	1.226	2.75
30	8.48	1.227	2.65
40	8.59	1.239	1.72